Growth of giant magnetoresistance spin valves using indium as a surfactant

W. F. Egelhoff, Jr., P. J. Chen, C. J. Powell, M. D. Stiles, and R. D. McMichael *National Institute of Standards and Technology, Gaithersburg, Maryland 20899*

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We have investigated the use of In as a surfactant to achieve smoother interfaces in spin-valve multilayers of the general type: $\text{FeMn/Ni}_{80}\text{Fe}_{20}/\text{Co/Cu/Co/Ni}_{80}\text{Fe}_{20}/\text{glass}$. The coupling field is reduced from ~ 0.8 to ~ 0.3 mT, presumably by suppressing roughness at the Co/Cu/Co interfaces, when 0.5-1.0 nm In is deposited on the first Co film just prior to Cu deposition or on the Cu film just prior to deposition of the second Co film. The In has a strong tendency to float-out to the surface during deposition of the spin valve leaving the spin-valve layers largely intact. The exchange bias at the FeMn/Ni $_{80}$ Fe $_{20}$ interface can be increased from 12 to 25 mT by the use of thicker In (1.4 nm). [S0021-8979(96)07604-6]

I. INTRODUCTION

In the few years since the giant magnetoresistance (GMR) effect was discovered, 1-3 much research has been directed at attempts to retain large GMR values while decreasing the size of the magnetic field required to produce the effect. Technological applications of great economic importance are likely to result if such efforts are successful. There does not appear to be any fundamental barrier, in the physics of the problem, preventing low saturation fields. If samples could be tailor made at the atomic level with atomic perfection it should be possible to reduce the saturation fields considerably. Atomic-scale engineering of the arrangement of atoms should make it possible to reduce contributions to the saturation field such as the coercivity, the anisotropy, and the magnetostatic coupling to almost arbitrarily low levels.

Therefore, the goal of achieving a large GMR at a low field will probably best be reached through the development of improved techniques for the control of atomic structure during thin-film deposition. One avenue for such improvement that has not been explored previously is the use of surfactant layers to modify film growth.

Several years ago it was suggested and successfully demonstrated⁴ that adsorbate layers which float-out or segregate to the surface during growth might be used to modify or control epitaxy in a favorable manner. In the few years since this discovery was made, there has been an extraordinary rapid development of this concept, primarily in the field of semiconductors,⁵ but also in metal-on-metal systems.^{4,6} Among the adsorbed species that have been investigated as surfactants are H, C, N, O, CO, and S,⁴ and As, Ag, In, Sn, Sb, Te, Pb, and Bi.^{5,6} A variety of favorable effects has been reported for surfactant-assisted growth, but the most common are improvements in the quality of interfaces by making them flatter, more coherent, less prone to interdiffusion, etc. However, so far no studies have appeared on surfactant-assisted GMR spin-valve growth.

In view of the key role that interfaces are thought to play in properties of GMR spin valves, it seems very appropriate to investigate whether surfactants can improve any of these properties. There are quite a number of species that could potentially act as surfactants in spin-valve systems. Moreover, the optimum conditions for surfactant-assisted spinvalve growth might well involve deposition of some segments of the spin valve at temperatures other than room temperature, and different surfactants might be best for different segments. Thus, a vast expanse of parameter space awaits exploration. The present article can only be considered a preliminary investigation of the application of one potential surfactant, indium, in the room temperature growth of one type of GMR spin valve.

Indium was chosen because it is a soft metal with a large atomic volume. In general, soft metals tend to exhibit rapid surface diffusion and low surface free energies, properties that favor their floating-out to the surface during overlayer deposition. The large atomic volume also favors the floating-out process since the incorporation of a large atom in a small lattice, such as In in Co or Cu, would cost a great deal of energy in the form of lattice strain.

II. EXPERIMENT

The substrates used in this work were 12-mm-diam cover-glass slides, cleaned ultrasonically in a glassware cleaning solution, rinsed in distilled water, dried, and installed in the deposition chamber. The base pressure before depositing a spin valve was typically 2×10^{-8} Torr ($\sim10^{-6}$ Pa) of which $\sim95\%$ was H_2 and the remainder largely H_2O . The presence of H_2 during deposition has no apparent effect on spin-valve properties unless the partial pressure approaches $\sim10^{-6}$ Torr. The low base pressure is achieved partly by depositing a ~1.5 nm Ti film on the inside of the deposition chamber from a centrally mounted Ti filament just prior to deposition of each spin valve.

It is very important to remove the hydrocarbon contamination (several tenths of a nm of which is accumulated on the glass substrate from exposure to the laboratory air) prior to the deposition of each spin valve in order to achieve the highest GMR values. Substrates were sputtered with a neutralized-beam Ar-ion gun at a beam energy of 500 eV until the carbon was removed, as judged by x-ray photoelectron spectroscopy (XPS) measurements in a connected vacuum chamber.

9 nm FeMn
2.5 nm Ni ₈₀ Fe ₂₀
2.1 nm Co
2.5 nm Cu
2.1 nm Co
5.5 nm Ni ₈₀ Fe ₂₀
// Glass slide //

FIG. 1. An illustration of the standard spin-valve structure that is the basis for the present investigations.

The metal films were deposited by dc-magnetron sputtering in 2 mTorr Ar at a rate of ~ 0.1 nm/s. During deposition, the samples were subject to an in-plane field of ~ 20 mT (200 Oe) provided by permanent magnets mounted on either side of the sample on two quartz-crystal-oscillator holders. The thickness of each metal film that is deposited is determined by the readings on the two quartz-crystal oscillators. The magnetoresistance measurements were made in the dc mode in yet another connected vacuum chamber using a four-probe with a 5 1/2 digit ohm meter. Values of the four-probe resistance can be converted into sheet resistance by multiplying by 4.1.

III. RESULTS AND DISCUSSION

The present work was based on a rather common type of spin-valve structure FeMn/Ni₈₀Fe₂₀/Co/Cu/Co/Ni₈₀Fe₂₀, which often achieves a moderate GMR at a rather low coercivity. The top two magnetic films (Co and Ni₈₀Fe₂₀) are pinned by exchange bias from the FeMn, and the bottom two magnetic films are free to switch at low applied fields (unpinned). Adjacent Co and Ni₈₀Fe₂₀ films are coupled so strongly that they always switch as a single magnetic unit. The standard sample of this type used as a reference point in the present work is illustrated in Fig. 1.

In a preliminary series of experiments, we determined the best point in the spin-valve structure for the deposition of

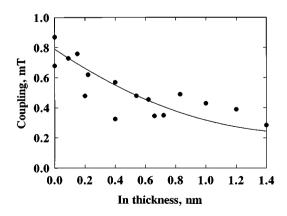


FIG. 2. A plot of the coupling field as a function of the average thickness of In deposited on the first Co film just before deposition of the Cu film. The solid line is a polynomial fit to the data. Note 1 mT = 10 Oe.

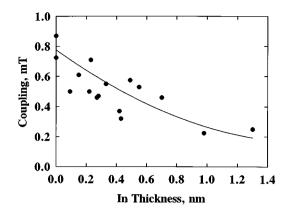


FIG. 3. A plot of the coupling field as a function of the average thickness of In deposited on the Cu film just before deposition of the second Co film. The solid line is a polynomial fit to the data. Note 1 mT=10 Oe.

In. The largest effects occurred when In was deposited either on the first Co film (just before the Cu film was deposited) or on the Cu film (just before the second Co film was deposited). This result is perhaps not surprising since the Co/Cu/Co layers are at the heart of the spin valve, and it is generally believed that the spin-valve properties are greatly dependent on the nature of these interfaces. The property that exhibited the largest change due to In was the coupling field that exists between the pinned and unpinned magnetic layers. It decreased by more than one-half due to In. Figures 2 and 3 present these data on the coupling field as a function of In thickness for In deposited on the first Co film and on the Cu film, respectively.

The most likely explanation for the reduction of the coupling field in Figs. 2 and 3 is that the In made the Co/Cu/Co layers smoother. For our samples of the type illustrated in Fig. 1, the coupling field is always ferromagnetic and is due primarily to the magnetostatic interaction across the Cu which follows from film roughness.⁸ The most important form of roughness is the long-wavelength roughness represented by the valleys between grains [as seen in our scanning tunneling microscopy (STM) images].^{9,10} This long-wavelength roughness should, according to Néel's model, make a major contribution to the coupling field. Figure 4 illustrates the Néel model. When two magnetic films are separated by a nonmagnetic film, any bumps or protrusions in the magnetic films will have magnetic poles on them, and

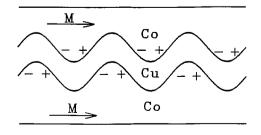


FIG. 4. An illustration of the "orange peel" coupling idea of Néel (Ref. 8) in which magnetostatic coupling occurs due to the interaction of magnetic poles in a magnetic/nonmagnetic/magnetic structure with conformal roughness.

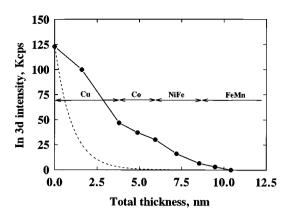


FIG. 5. A plot of the In 3d x-ray photoelectron intensity for 0.5 nm In deposited on the first Co film as a function of the thickness of subsequently deposited layers of Cu, Co, $Ni_{80}Fe_{20}$, and FeMn. The solid line connects the data points. The dotted line is the decrease in intensity predicted on the basis of photoelectron escape depths if there were no surface segregation of In.

a dipole field will be set up (this model assumes that the magnetization is in the plane of the film). If this roughness is conformal (i.e., if the same bumps occur in all three films one above another), then the dipole fields will interact in a manner that tends to produce parallel (or ferromagnetic) alignment in the magnetic films. This effect was termed "orange peel" coupling by Néel.⁸

Several simple principles may be inferred from Fig. 4. First, the steeper the slopes, the greater the magnetic pole density; hence the stronger the coupling. Second, the closer the peaks and valleys are to one another (e.g., the smaller the grain size), the stronger the coupling will be. Finally, the thinner the Cu film, the larger the coupling will be.

Our STM results generally confirm the concept that rougher samples (those with deeper grain boundary valleys) tend to exhibit larger coupling fields than smoother ones. Moreover, we have calculated the values of the coupling field (using the slopes in the STM images to determine the magnetic pole densities and summing over the dipolar interactions). The calculated couplings are generally within about 20% of the observed values. 9 This agreement suggests that the coupling is indeed magnetostatic. Furthermore, the coupling fields for our standard samples are almost identical in measurements made at 150 K and at room temperature. This independence of temperature would be expected for a magnetostatic effect. We have not found any evidence of oscillatory coupling in our standard samples (grown at room temperature) of the type illustrated in Fig. 1. We find the coupling is always ferromagnetic and rises monotonically with decreasing Cu thickness. From this evidence we conclude that the oscillatory coupling often found in GMR superlattices does not play a noticeable role in our samples.

Figure 5 presents data from XPS on the surface segregation or floating-out of In as the layers of a spin valve are deposited. In this case, a film of In 0.5 nm thick (on average) was deposited on the first Co film, and the decline in the intensity of the In 3d peaks was monitored during deposition of the Cu, Co, Ni₈₀Fe₂₀, and FeMn overlayers. For In, 0.5 nm corresponds to roughly 2 monolayers (ML). The decline

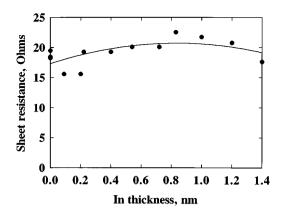


FIG. 6. A plot of the sheet resistance of the GMR spin valve vs the thickness of In deposited on the first Co film.

with increasing overlayer thickness is due to the incomplete segregation of In, i.e., it gradually gets left behind, trapped in the spin-valve layers.

The dashed curve in Fig. 5 indicates the decline in the In 3d intensity that would be expected on the basis of photoelectron escape depths if no In surface segregation occurred. A comparison of the dashed curve with the data suggests that roughly half of the In (0.25 nm, or 1 ML) is left behind in the first 2.5 nm of Cu (\sim 14 ML of Cu), implying an average In concentration of about 7 at. An alternative way to view this result is that after each ML of Cu is deposited, \sim 95% of the In that had been at the surface is still at the surface (0.95¹⁴=0.5). The thermodynamic driving forces for this segregation are the larger atomic volume of In which strains the Cu lattice upon incorporation and the lower surface free energy of In.

The mechanism of surface segregation is likely to involve surface diffusion of In and Cu atoms. The rate of diffusion of Cu adatoms and vacancies at room temperature on Cu surfaces is very high. ¹² Since In is a softer metal than Cu, it should experience an even higher rate of diffusion, and single-crystal studies seem to bear this idea out. ¹³ Thus, the atoms on the In/Cu surface are extremely mobile at room temperature. In this active environment, deposited Cu atoms will find their way to the substrate, with which they are well lattice matched and will bond there. The In film, which is very poorly lattice matched to the substrate must be very disordered and may even be a near-liquid-like overlayer.

An additional indication of the incorporated In is the increase in the resistivity of the sample. Figure 6 presents the sheet resistance of the spin valve as a function of the thickness of In deposited on the first Co film. The sheet resistance increases from 17 to 20 Ω/\Box for a 0.5 nm In thickness. In terms of bulk resistivities, these values become 48 and 56 $\mu\Omega$ cm. These values are large because the high-resistivity alloys FeMn and Ni $_{80}$ Fe $_{20}$ account for much of the total thickness. The increase in resistivity due to the use of In is probably due mostly to the In incorporated in the Cu, an alloy for which increases of 1.1 $\mu\Omega$ cm/at. % In are reported. ¹⁴

Two effects of the incorporation of In in Cu that may be expected are an increase in electron scattering in Cu and a

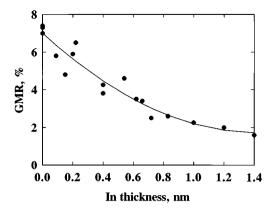


FIG. 7. A plot of the GRM vs the thickness of In deposited on the first Co film just before deposition of the Cu film.

consequent reduction in GMR. Figures 7 and 8 present the measured GMR versus the average thickness of In deposited on the first Co film and on the Cu film, respectively. This loss of GMR is an important shortcoming in the use of In as a surfactant. In future work, we plan to investigate various approaches to reducing the In incorporation, including post-deposition annealing and deposition at elevated temperatures.

Our interpretation of the reduced coupling field in terms of smoother interfaces might be questioned on the basis that the incorporated In increases the effective thickness of Cu and that this increased thickness should reduce the magnetostatic coupling. However, Fig. 9 presents data suggesting that this increased thickness does not account for the reduction in the coupling field observed in Figs. 2 and 3. A general principle of magnetostatic coupling is that, for a given roughness, the strength of the magnetostatic coupling field is largely determined by the thickness of the nonmagnetic spacer layer, and the composition of the nonmagnetic spacer layer is almost irrelevant. In Fig. 9, an additional 0.25 nm Cu (the thickness of the incorporated In, as implied by Fig. 5) reduces the coupling field by a negligible amount. Therefore, smoother interfaces due to a surfactant effect of In constitutes a more plausible interpretation for the reduced coupling field.

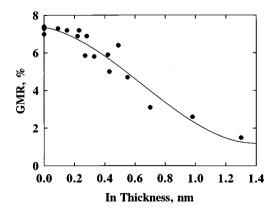


FIG. 8. A plot of the GMR vs the thickness of In deposited on the Cu film just before deposition of the second Co film.

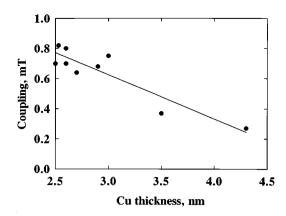


FIG. 9. A plot of the coupling field vs the thickness of the Cu film. Note 1 mT=10 Oe.

One very striking positive effect of In occurs in the exchange bias that the FeMn provides to pin the top magnetic films. Figure 10 presents the dependence of this exchange bias field on the thickness of In deposited on the first Co layer. The effect is small for 0.5 nm In, presumably because (as Fig. 5 indicates) very little In remains on the surface when the FeMn is deposited. However, for thicker In films (when presumably more In is present), a marked increase in the exchange bias is observed. This observation suggests that future research should consider the use of surfactants as a means of increasing the exchange bias.

One property that does not change as a function of In thickness is the coercivity of the unpinned layer. Figure 11 presents the data. As may be expected, the coercivity of the unpinned films is not influenced by the In deposited on the first Co film since nearly all of the In segregates onto the Cu.

IV. CONCLUSIONS

The major conclusions of this work are as follows:

- (1) Indium can be used as a surfactant in GMR spinvalve systems, improving some surface and interfacial properties but impairing others.
- (2) The magnetostatic coupling decreases due to the use of In, apparently because of smoother Co/Cu/Co interfaces.

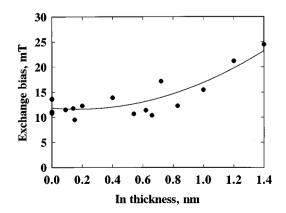


FIG. 10. A plot of the exchange bias vs the thickness of In deposited on the first Co film. Note 1 mT=10 Oe.

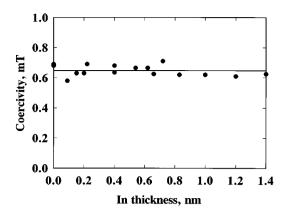


FIG. 11. A plot of the coercivity of the unpinned films vs the thickness of In deposited on the first Co film. Note 1 mT=10 Oe.

- (3) The GMR declines due to the incorporation of \sim 7 at. % In the Cu.
- (4) The exchange bias at the FeMn/Ni $_{80}$ Fe $_{20}$ interface increases due to the use of In.
- (5) Future work on surfactants in spin-valve systems should explore the use of higher substrate temperatures during deposition to reduce the incorporation of In in the Cu.

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